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Influence of cationic polyacrylamide on three-dimensional fluorescence analysis in water treatment by coagulation

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ABSTRACT

Removal of dissolved organic matter (DOM) is a major problem in drinking water treatment, as its presence affects color, taste, and odour and during disinfection, carcinogenic by-products can be created. To provide the best DOM removal, detailed characterisation is required and the combination of three-dimensional fluorescence excitation-emission matrix spectroscopy and parallel factor modelling has become a popular approach. However, some components in the system may affect the characterization. We tested the influence of popular coagulants, polymeric ferric sulfate and cationic polyacrylamide on the three-dimensional fluorescence excitation-emission wavelength spectra of DOM during coagulation treatment. The parallel factor model revealed 3 fluorophore components (C1, C2, and C3). The organic polymer (cationic polyacrylamide) has a negative influence on monitoring, significantly reducing the uncorrected matrix correlation between fluorescence and DOM for the three components from 0.970, 0.976 and 0.906 to 0.941, 0.944 and 0.617, respectively, and also the linear correlation coefficients for the C1 and C2 from 0.92 and 0.84 to 0.83 and 0.79, respectively. The C3 component, a protein-like material, was the most affected. The results demonstrate the need to consider residual polymer as a potential interference when using fluorescence to assess the degree of success of DOM removal by coagulation.

KEYWORDS: Coagulation; dissolved organic matter; three-dimensional fluorescence; cationic polyacrylamide

1 INTRODUTION

The treatment of organic matter in aqueous systems has been an important research topic in applied environmental science (Jain et al. 2018, Nidheesh 2018, Wei Wang 2018, Xiaocun Zhuo 2019). Coagulation is an important operational unit in the water treatment process and is a preferred method for the removal of dissolved organic matter (DOM) in the production of drinking water (Dennett et al. 1996, Eric Lichtfouse 2019, Hua et al. 2016, Matilainen et al. 2010, Zhu et al. 2016). In particular, its application as a pre-treatment step to protect membrane based treatment systems where the presence of DOM results in membrane fouling (Bu et al. 2019, Tang et al. 2010). The evaluation of its effectiveness is usually carried out by measuring dissolved organic carbon (DOC) and ultraviolet absorption value at 254 nm (UV_{254}, cm^{-1}). Whilst this provides an assessment of DOM concentration, knowledge of other characteristics is needed for effective treatment (Hua et al. 2017, Hua et al. 2018, Sanchez et al. 2014). More recently, three-dimensional fluorescence excitation emission matrix spectroscopy has been developed for the evaluation of engineering systems for water treatment (Carstea et al. 2016, Zhu et al. 2017). It is a powerful tool, which is able to capture the specific components of DOM through a simple operational process. As a promising technology for characterization and tracking of DOM, it has also been coupled with parallel factor modelling in assessing coagulation systems (Yang et al. 2015b, Zhu et al. 2017).

In water treatment, large amounts of inorganic metal salt coagulants are used, for example poly aluminum chloride and poly ferric sulfate. Two types of coagulation are typically used: inorganic, un-hybridized coagulation using an inorganic coagulant alone, and inorganic-organic hybrid coagulation with mixed organic and inorganic coagulants (Lee et al. 2012, Zhu et al. 2011). For hybrid coagulation, cationic

polyacrylamide (C_3H_5NO) is a common substance, which has a strong fluorescence signal (Zhu et al. 2017). The cationic polyacrylamide is a linear high molecular weight polymer, with a number of functional groups and has the potential to form hydrogen bonds with various substances. Its use reduces the coagulant dose particularly when dealing with a high turbidity in water treatment systems. Most of the cationic polyacrylamide added is removed by sedimentation along with the treatment floc, but there is potential for residual polyacrylamide to remain in the treated water. The cationic polyacrylamide is not toxic in water but its monomer acrylamide is known to be harmful to humans (Zhu et al. 2013). Low concentration cationic polyacrylamide and acrylamide are difficult to measure, and until now, the presence of cationic polyacrylamide in treated water is not clear. In coagulation treatment systems, the analysis of the variation of DOM characteristics has been approached using excitation emission matrix spectroscopy and parallel factor models (Jaffé et al. 2014, Yang et al. 2015a).

In the study reported here, an assessment of the three dimensional fluorescence of DOM was undertaken by coupling excitation-emission matrix spectroscopy with a parallel factor model. The influence of the cationic polyacrylamide on the coagulation of DOM was investigated by measuring similarities between components, and correlation coefficient (r) with DOC. A comparison undertaken between raw water, and samples treated by hybrid and un-hybridized coagulation. The un-hybridized coagulation used a polymeric ferric sulfate coagulant, while the hybrid coagulation used two coagulants polymeric ferric sulfate and cationic polyacrylamide.

2 MATERIALS AND METHODS

2.1 Materials

The polymeric ferric sulfate was prepared in our laboratory using analytical grade materials: ferrous

sulfate heptahydrate (Sinopharm Chemical Reagent Beijing Co., Ltd., China), sodium acetate (Sinopharm Chemical Reagent Beijing Co., Ltd., China), zinc sulfate (Tianjin Damao Chemical Reagent Co., China), hydrochloric acid (Zhuzhou XingKong Huabo Co., Ltd., China), sodium chlorate (Tianjin Kemiou Chemical Reagent Co., China), sodium carbonate (Tianjin Institute of Chemical Reagents, China), concentrated sulfuric acid (Zhuzhou XingKong Huabo Co., Ltd., China). Commercial cationic polyacrylamide (Zouping Mingxing 119 Chemical Co. Ltd., Binzhou, China) was used in this study without further purification. All aqueous solutions and standard solutions used in this study were prepared with deionized water. The glassware and other labware were acid-washed, rinsed thoroughly with water, and dried prior to use.

2.2 Jar test

A water sample was collected from an artificial lake (Yuehu lake) located on the campus of Hunan University of Science and Technology, China. A coagulation-flocculation experiment carried out using a program-controlled jar test apparatus (ZR4-6, ZhongRun Water Industry Technology Development Co. Ltd., Shenzhen, China) at room temperature. Each experimental run used 1 L water samples transferred into a beaker. After adding individual coagulants or double coagulants the sample was rapidly mixed at a set agitation speed (rpm) for 2 min, followed by a slow mixing phase at 70 rpm for 15 min, and then a 30 min settling time. The supernatant sample was extracted from the beaker 2 cm below the water surface to measure water quality parameters.

2.3 Excitation-emission matrix spectroscopy collection

The excitation-emission spectra of 0.45 μm pore size membrane filtered water samples were tested using an FL-4600 fluorescence spectrophotometer (Hitachi High Technologies, Tokyo, Japan). The filtrates

were collected for DOC measurement using an Elementar Vario MICRO Cube (Elementar Analysensysteme GmbH, Germany). The excitation wavelengths and emission wavelengths were fixed from 200 nm to 400 nm in 2 nm steps, and from 200 to 500 nm in 3 nm steps, respectively. The fluorescence intensity of an ultra-pure water as a blank sample was subtracted. The Rayleigh and Raman scatter were removed following the reported procedure as found in (Bahram et al. 2006). The corrected excitation-emission spectroscopy data was fractioned into different components using the parallel factor model (Moradi et al. 2018). The split-half analysis was used to identify the best number of the factors. The maximum fluorescence intensity (F_{max}) of each derived component was used to evaluate quantitative and qualitative differences between samples. The parallel factor model analysis was performed on MATLAB using the N-way toolbox (Andersson and Bro 2000).

2.4 Uncorrected matrix correlation

Quantitative similarities between DOM fluorescence fractions of raw water and treated samples were evaluated using a validation of uncorrected matrix correlation (UMC). The correlation evaluates the degree of similarity between two-component fluorescent mixtures through the sum of the eigenvalues of the m x n matrices using the following equation (Burdick and Tu 1989, Sanchez 2013):

$$UMC(A,B)=Trace(A^TB)/\|A\|_F\|B\|_F$$

where A and B are the matrices under comparison. $\|A\|$ is $[trac(A^TA)]^{1/2}$, and $\|B\|$ is $[trace(B^TB)]^{1/2}$.

3 RESULTS AND DISCUSSION

3.1 Fluorescence spectra of cationic polyacrylamide and polymeric ferric sulfate

Scanning data of the excitation-emission matrix spectra of cationic polyacrylamide and polymeric ferric sulfate is shown in Fig. 1. As illustrated in Fig. 1a, the results showed that the cationic polyacrylamide

showed two characteristic peaks, positioned in excitation/emission wavelength about 230 nm/340 nm and 280 nm/340 nm, respectively. These peaks belong to protein-like materials resulting from nitrogen-containing groups (Zhu et al. 2017). As shown in Fig. 1b, the polymeric ferric sulfate did not display any characteristic peak in the range of emission wavelength from 300 nm to 400 nm. Therefore, the fluorescence of the organic cationic polyacrylamide coagulant is significantly different from the poly ferric sulfate.

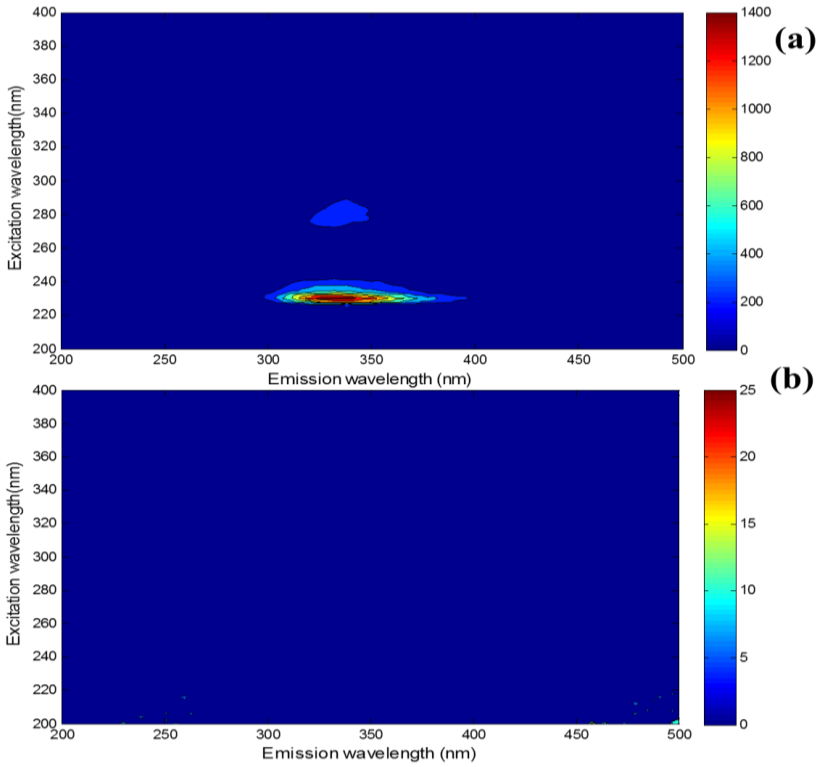

Fig. 1 Three dimension fluorescence excitation-emission matrix spectra of (a) cationic polyacrylamide at 0.06 g/L and (b) polymeric ferric sulfate at 3.3 g/L. This highlights the fluorescence signal background for the organic coagulant.

3.2 Spectroscopic comparison

We randomly selected sample spectra of raw water, un-hybridized coagulation and hybrid coagulation (Fig. 2). The dissolved organic carbon (DOC) concentration in raw water was 15 mg/L and pH was

around 7. We found that the spectrum of raw water was similar to that from inorganic coagulation, but the sample changed greatly after hybrid coagulation (see Fig. 2a). These results showed that the presence of cationic polyacrylamide could lead to a significant change in the spectroscopic properties of the sample. We quantitatively studied the similarity of the matrix data using UMC. The UMC value ranged between 0 and 1 and was used to measure the degree of spectral overlapping between two components (Burdick and Tu 1989). The 0 and 1 values for matrices indicate null and complete spectral overlapping, respectively. The results showed that the value for UMC between the raw sample and the hybrid sample is 0.850 while that between raw sample and un-hybridized sample is 0.957. The hybrid coagulation generated a more significant difference in excitation-emission matrix spectra from that of raw sample and un-hybridized sample. At the excitation wavelength of 230 nm, new peaks were also formed in the hybrid samples (see Fig. 2b). We measured the spectroscopy of cationic polyacrylamide at emission wavelength of 401 nm, and found that there are three significant peaks around 205, 230 nm and 280 nm (see Fig, 2c), corresponding approximately to ultraviolet adsorption peaks of some monomers in polyacrylamide (Sun et al. 2014). Also we measured the samples after coagulation which showed that except for the sample from hybrid coagulation, the other samples no significant peaks was observed around 205 nm, 230 nm and 280 nm, suggesting that the residual cationic polyacrylamide remained in water (see Fig, 2c).

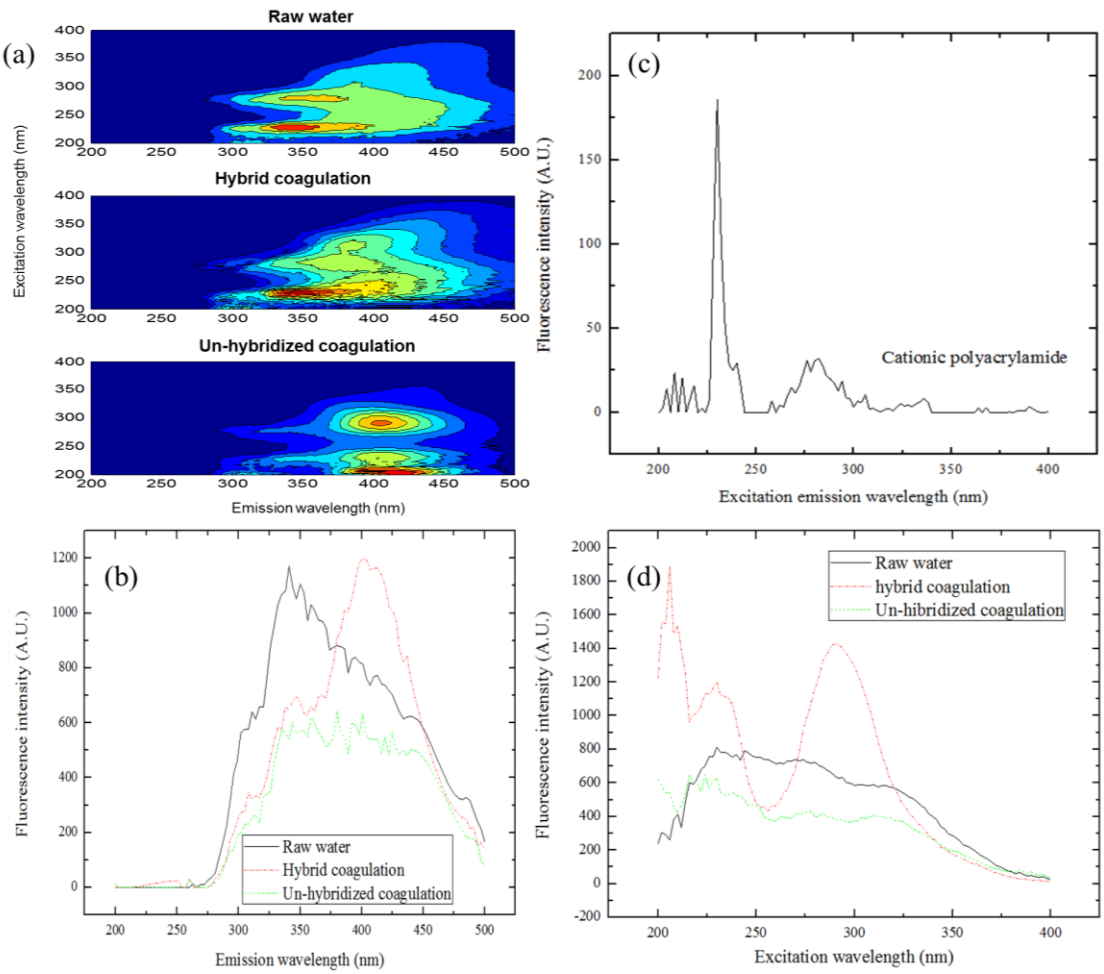

Fig. 2 (a) Excitation-emission spectra of raw water, un-hybridized coagulation sample and hybrid coagulation sample, (b) emission wavelength variation at excitation wavelength of 230 nm, (c) fluorescence spectroscopy of cationic polyacrylamide at excitation wavelength of 401 nm, and (d) fluorescence spectroscopy of coagulation samples at emission wavelength (nm) of 401 nm. The results showed that the cationic polyacrylamide remains in the treated water thus masking the fluorescence of DOM.

3.3 Comparison of parallel factor model components

Parallel factor model assessment of DOM in natural and engineered systems has been a high priority and an assessment method has been evaluated (Sanchez et al. 2014). The excitation-emission spectroscopy can identify its components and monitor changes. However, the feasibility of a parallel factor model

assessment of DOM for hybrid inorganic-organic coagulation systems has yet to be addressed. Therefore, we used parallel factor model to investigate the characteristics of DOM components in coagulation. In our work, we made a comparison between parallel factor model components in raw water and treated water. Three independent parallel factor model components (denoted as C1, C2 and C3) were identified: for the raw water sample (R-C1, R-C2, and R-C3); in hybrid coagulation system (H-C1, H-C2, and H-C3); in un-hybridized coagulation systems (UH-C1, UH-C2, and UH-C3). The C1 component shows two excitation maxima at 232 and 288 nm with a single emission maximum at 392 nm, which has been identified as a humic-like fluorophore (Ishii and Boyer 2012). For C2, an excitation maximum at a wavelength below 266 nm and a secondary excitation peak at 342 nm with a maximum emission at 452 nm, which has been classified as a humic-like fluorophore (Ishii and Boyer 2012). The C1 is (marine) and terrestrial humic material resulting from possible microbial reprocessing, while C2 is a terrestrially-derived component (Piotr Kowalczuk 2009). The C3 had two main excitation maxima at 226 nm and 276 nm, with an emission peak at 332 nm. They all resemble protein-like structures similar to tryptophan (Piotr Kowalczuk 2009). Extracted fluorescence components showed similar fluorescence characteristics as seen from reoccurring parallel factor model components (see Figs. 3a–i) and their spectral loadings (see Figs. 3j and r). Fig. 3j to r show the excitation (denoted as Ex.) and emission (denoted as Em.) loadings for each component, obtained from parallel factor model on random halves of the data array.

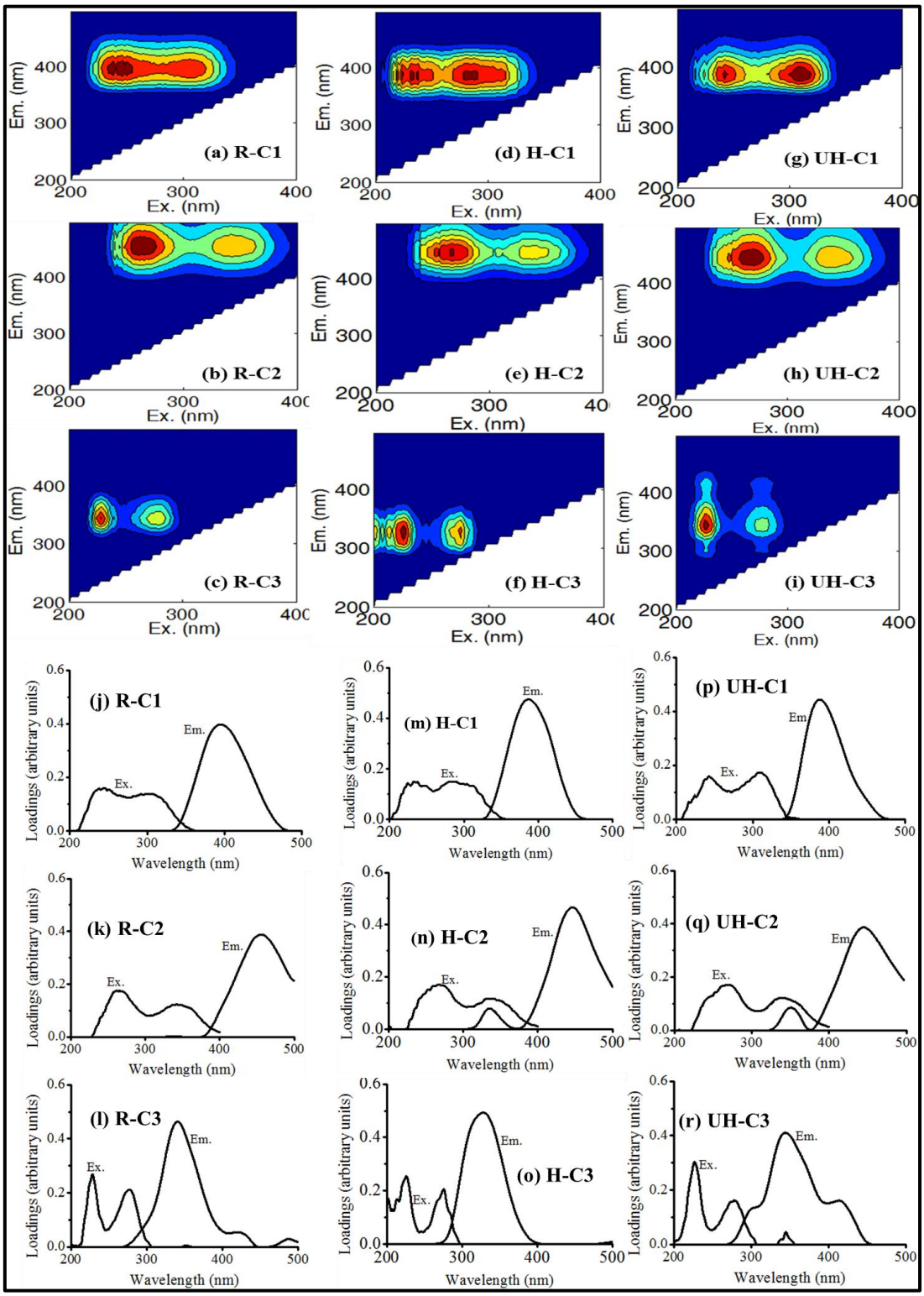

Fig. 3 Signatures of three parallel factor model components (a-c; j-l) identified in raw water, in hybrid coagulation (d-f; m-o) and in un-hybridized coagulation (g-i; p-r). Contour plots show spectral shapes of excitation and emission of derived components (a-i). Excitation and emission loadings (j-r) on the left are denoted as Ex., and on right side, they are denoted as Em. Samples were collected from raw water,

hybrid coagulation and un-hybridized coagulation, and input into the parallel factor model. It showed that three components were identified in both of raw water and treated water.

3.4 Quantitative similarity analysis

The UMC values of R-C1 and H-C1, R-C2 and H-C2, as well as R-C3 and H-C3, were 0.941, 944 and 0.617, respectively. C1, and C2 had a relatively complete spectral overlapping but C3 had a significant deviation and H-C3 and R-C3 were not comparable. New spectral loadings between 200 nm and 226 nm in treated samples were found, which belong to protein-like materials. However, we found that in un-hybridized coagulation, the deviation of spectral overlap between R-C3 and UH-C3 was low, and the spectral loadings also disappeared. The measured results showed that the UMC values of UH-C1 and R-C1, UH-C2 and R-C2, as well as UH-C3 and R-C3 were 0.970, 0.976 and 0.906, respectively. Except for the groups (UH-C2 and R-C2, UH-C1 and R-C1), the UH-C3 and R-C3 were also comparable. The UMC values were higher than those in hybrid coagulation. This showed that the components in un-hybridized coagulation were comparable with the components found in raw water. In the un-hybridized coagulation system, assessment of the protein-like material was not possible. These changes in peak regions were consistent with characteristic peaks for the cationic polyacrylamide, suggesting residual components post treatment.

3.5 Correlation analysis

Good relationship between DOM components of parallel factor model and water quality parameters such as DOC, during coagulation treatment, has been widely recognized (Aftab and Hur 2017, Sanchez et al. 2013, Zhu et al. 2014). The correlation of components with DOC was evaluated using correlation coefficient, *r*. As shown in Fig. 4, the maximum fluorescence intensity (F_{max}) the removal (%) of components (C1 and C2) and removal of DOC (%) correlate well (r = 0.92 and r = 0.83) with C1 in un-

hybridized coagulation and hybrid coagulation, respectively; hence, r = 0.84 and r = 0.79 for C2 in un-hybridized coagulation and hybrid coagulation, respectively. However, un-hybridized coagulation had a better effect than hybrid coagulation. It implied that the reliability of parallel factor model assessment in DOM removal for hybrid coagulation was reduced.

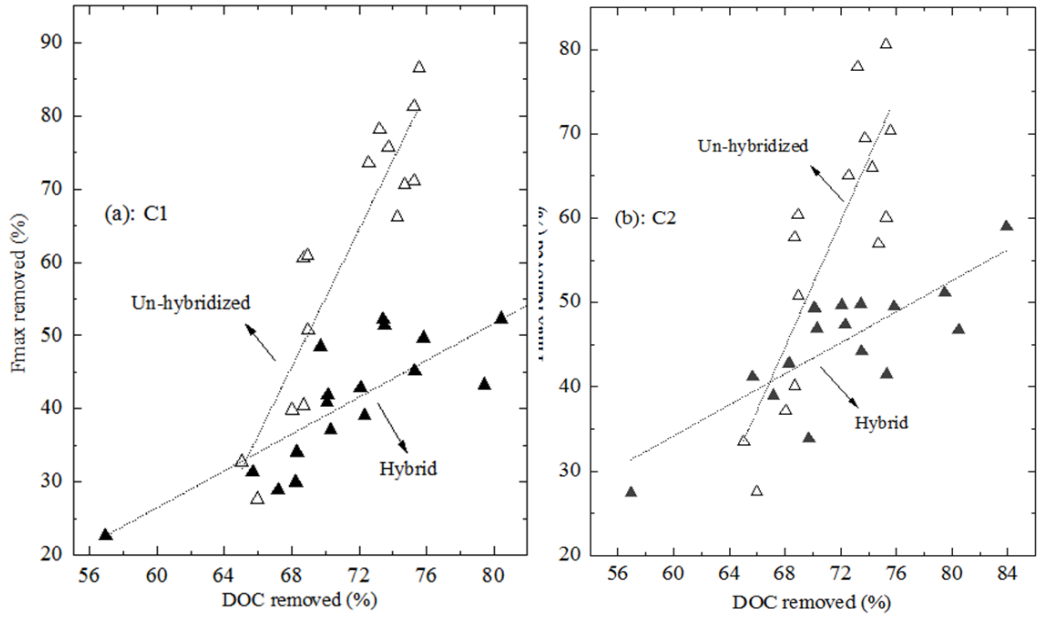

Fig. 4 The correlation results of F_{max} removal (%) of (a) C1and (b) C2 with DOC removal (%). The correlation results showed that the *r* values were 0.92 and 0.83 in un-hybridized and hybrid coagulation for C1, respectively, and 0.84 and 0.79 in un-hybridized and hybrid coagulation for C2, respectively, showing that the presence of cationic polyacrylamide decreased the linear correlation between water quality with fluorescence components.

4. CONCLUSIONS

We have studied the influence of the cationic polyacrylamide on three-dimensional fluorescence analysis of DOM during coagulation treatment processes. The results showed that the presence of the cationic polyacrylamide in residual processes results in adverse effect in the analysis of the results from hybrid coagulation and from un-hybridized. Three components (C1, C2 and C3) in hybrid coagulation samples

were compared with that from raw water, which showed lower UMC values but the C3 component could not be correlated. In addition, lower correlation coefficients were found between them and DOC, due to the presence of cationic polyacrylamide. This raises the issue that fluorescence excitation-emission matrix spectroscopy is unreliable when investigating DOM component variations without considering the effect from cationic polyacrylamide in hybrid coagulation treatment.

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